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Lyophilization for Water Recovery III, System Design

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ABSTRACT

Mixed liquid/solid wastes, including feces, water processor effluents, and food waste, can be lyophilized (freeze-dried) to recover the water they contain and stabilize the solids that remain. Our previous research has demonstrated the potential benefits of using thermoelectric heat pumps to build a lyophilizer for processing waste in microgravity. These results were used to build a working prototype suitable for ground-based human testing. This paper describes the prototype design and presents results of functional and performance tests.

INTRODUCTION

On the International Space Station and future missions, recovering potable water from wastewater is essential, and a variety of technologies have been evaluated for this purpose. In addition to potable water, every water processor produces its own waste stream – usually a concentrated aqueous solution, such as brine from distillation or reverse osmosis, or sludge from a bioreactor.

Water also plays a critical, though less obvious, role in solid waste processing. The presence of small amounts of water in solid waste complicates processing and storage. Many wastes are stable when dry but biologically or chemically unstable when wet: feces, toilet tissue, EVA diapers, sanitary napkins, food waste, vomitus, etc. For example, NASA's International Space Station Waste Collection System (US ISS WCS) collects feces and toilet paper in porous bags, which are then stored in fiberglass canisters. The canisters are vented to cabin atmosphere, to prevent pressurization by carbon dioxide and other gases generated during fecal biodegradation. A disposable activated carbon filter built into each canister scrubs some, but not all, malodorous hydrocarbons from this gas stream; the balance is released to the cabin, along with the carbon dioxide. If this waste were dried, it could simply be stored in airtight bags. The water released could be fed to the water processor and reused.

Mixed liquid/solid wastes are problematic for both wastewater and solid waste processors. This category includes processor effluents such as brines and pyrolyzed organic solutions, and processor influents such as solid waste destined for compaction, particle size reduction, or simple storage. Most wastes in this category contain odor-causing compounds or pathogens, so finding a way to handle and store them is of particular importance.

A dryer fills the gap between wastewater and solid waste processors by separating water from solids. But the category of wastes that would benefit from drying is diverse, and includes materials that are biologically active, rich in volatile organics, and pose material handling problems. So the ideal dryer would 1) minimize transfer of volatiles other than water during drying, 2) minimize chemical and biological reactions during drying, and 3) handle both liquids and solids in microgravity.

Lyophilization addresses goals 1) and 2) exceptionally well, and also offers benefits with respect to goal 3). Lyophilization can be thought of as two separation processes in one – it is essentially freeze separation followed by vacuum distillation. Material is first frozen, separating it into a matrix of nearly pure ice crystals interspersed with regions of crystalline or glassy phases low in water. Then moderate vacuum (approximately 100 Pa) is applied and the ice crystals vaporize; this water vapor condenses on an adjacent cold surface. Volatilization of organics from the other phases is impeded, because the dense web of organics in these solid phases presents a mass transfer limitation. Freezing before drying eliminates problems associated with maintaining a free liquid-vapor interface in microgravity, and prevents case hardening – the formation of a surface "soup skin" of concentrated, viscous solution that can stall drying of some materials.

BACKGROUND

HEAT PUMPS – During conventional drying or lyophilization, the phase change of liquid water or ice to water vapor removes heat from the remaining solid. This lowers the temperature of the sample and the vapor pressure of its remaining water. For drying to continue, heat must be added to the sample from an external source. Similarly, heat must be removed from the cold surface on which the water vapor condenses. A heat pump connected between the sample and condenser can perform both these functions, recycling the heat. Heat pump energy efficiency will be a function of the temperature difference between its hot side (sample) and cold side (condenser). The thermodynamic efficiency limit is that of a Carnot cycle:

$$\mathbf{COP} = \frac{\mathbf{Q_c}}{\mathbf{W}} = \frac{\mathbf{T_c}}{\mathbf{T_h - T_c}} \qquad (1)$$

COP is the coefficient of performance, the ratio of heat removed from the cold side (energy desired) over work to drive the pump (energy that costs). At low temperature differences, COP can be quite high – e.g., for potential lyophilizer conditions of T_c = 260K and $T_h - T_c$ = 10K, COP = 26. The temperature difference necessary to drive drying at a given rate varies with sample geometry and sample material heat and mass transfer characteristics, as discussed below.

Typical heat pumps, which pump a working fluid through the Rankine cycle (slightly less efficient than Carnot), can perform near their theoretical COP under certain conditions. But if used in this context – pumping heat directly from condenser to sample during drying – engineering limitations hinder their practical efficiency. For example, heat transfer resistances within the working fluid limit a heat pump's ability to perform at very low temperature differences. Also, pipes that contain the pressurized fluid represent a significant heat flow pathway, resulting in heat leaks and insulation losses, particularly in smaller systems [1].

Thermoelectric heat pumps (TECs) have much lower theoretical efficiency than traditional heat pumps [2], but in a lyophilizer they can perform more efficiently in practice, because their simple design avoids the efficiency-reducing limitation of traditional heat pumps. The main practical efficiency limit common to systems using TECs is unwanted convective heat flow across the narrow gap between their hot and cold surfaces. Evacuating this gap virtually eliminates this heat leak, and lyophilization occurs under vacuum. As described previously [3, 4], a working thermoelectric lyophilizer can perform near its theoretical efficiency limit, provided it is well-engineered.

GENERAL DESIGN CONSIDERATIONS – Figure 1, adapted from [3], shows the basic configuration of a thermoelectric lyophilizer, and the flow of heat and water vapor during drying.

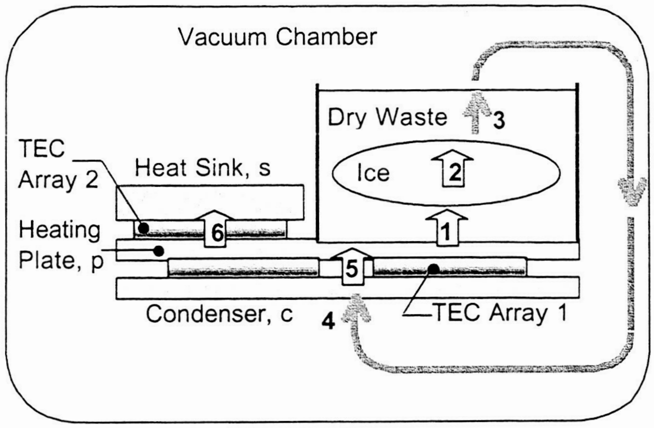

Figure 1. Simple thermoelectric lyophilizer schematic showing heat and mass flows

Numbered arrows indicate the following heat and mass flows:

1. Heat flows through dried sample material (if present) to ice that remains.
2. Heat flows through the ice to these surfaces where vaporization occurs, supplying the heat of sublimation.
3. Water vapor flows down a pressure gradient, through dried sample material, out to the vacuum chamber.
4. Water vapor deposits on the condenser, and its heat of sublimation flows through the existing ice layer.
5. Heat is pumped from the condenser to the heating plate.
6. Waste heat (Joule heating of the TECs in step six) is removed to an external heat sink through a second set of TECs.

Waste heat (generated by the TECs in step six) is removed to an external heat sink through a second set of TECs.

The efficiency of this system is a strong function of the temperature differences experienced by the TECs associated with arrows 5 and 6. The temperature difference across arrow 5 is a function of the resistance to heat and mass flow in arrows 1-4. The minimum temperature difference required across arrow 6 depends on the eutectic temperature (freezing point) of material being dried.

Figure 2, adapted from [4], represents the components in Figure 1 using an electrical circuit analogy (temperature = voltage, heat flow = current, etc.). The analogy provides a simple means of visualizing the significant heat and mass transfer pathways in the system, and serves as a tool for analyzing system performance and estimating the impact of design decisions on energy efficiency.

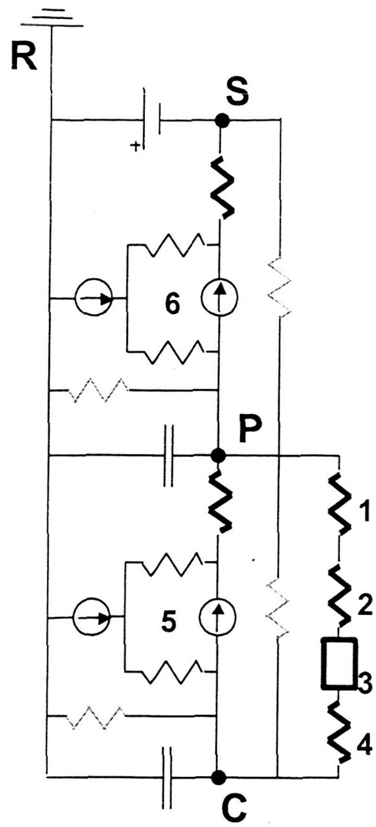

Figure 2. Electrical circuit analogy used for heat and mass transfer analysis. Numbers correspond to components in Figure 1. Efficiency improves as bold black resistors become small and gray resistors become large.

Resistors 1-4 represent the core process – heat and mass flow through the sample and condenser. (A box represents 3 because vapor transfer is a nonlinear function of temperature.) Resistors and current sources surrounding 5 and 6 represent the behavior of the TECs. Gray resistors are heat leaks – unwanted heat transfer pathways between components at different temperatures. The two unnumbered black resistors represent the resistance to heat transfer at the TEC surfaces and within the metal components themselves.

The circuit shows the impact of design decisions on system performance and energy efficiency. Efficiency can be improved by lowering the resistance to heat and mass transfer through 1-4, reducing the temperature difference between P and C. The most straightforward way to accomplish this is by minimizing sample thickness.

Experiments with the previous thermoelectric lyophilizer [4] have shown that resistance to heat and mass flow through the sample can be decreased dramatically by compressing the sample as it dries. This reduces both the thickness and porosity of the dry layer that forms between the sample and heat source, reducing resistance to heat transfer. Compressing the sample affects the rehydration characteristics of the dried product, but this is of no importance when drying waste to recover water.

Efficiency can also be improved by reducing heat leaks. If resistances through the gray resistors in Figure 2 are increased, less heat will leak between different components, reducing the load on the TECs and improving efficiency. At the low pressures under which drying occurs, convective heat transfer via the gas in the chamber is negligible. Heat leaks occur via conduction through solid connections between components, and to a lesser extent via infrared radiation.

APPLICATION-SPECIFIC DESIGN CONSIDERATIONS – Designing a working lyophilizer requires compromising between the energy efficiency goals described above and practical considerations associated with a specific application.

Of the list of wastes produced on human space missions that would benefit most from lyophilization, water processor effluent and feces have the highest estimated generation rates. Estimated flow rates for the Mars Reference Mission are listed in Table 1. Water processor effluent flow rate can be expected to vary significantly depending on type of water processor and wash water flow rate specified for a particular mission. In this case it represents approximately 80% of total water recoverable by lyophilization.

Table 1. Candidate Wastes, Mars Reference Mission, assumes water processor recovery rate of 99%. [5]

Waste Stream	Mass (wet) kg/day	Water %	Mass Water kg/day
Water processor effluent	3.05	88	2.69
Feces and toilet paper	0.86	63	0.54
Combined	3.91	83	3.23

But the other benefit of lyophilization, stabilization of solids, is more pressing in the case of feces than for water processor effluent. Also, collection of feces in microgravity is a nontrivial task requiring specialized hardware, an example of which is shown in Figure 3. And while water processor effluent is a liquid easily pumped through pipes, semi-solid toilet wastes presents more complex material handling problems. For these and other reasons, lyophilizing feces presents a higher design hurdle. The system described below was designed with an eye towards interfacing with a feces collection system.

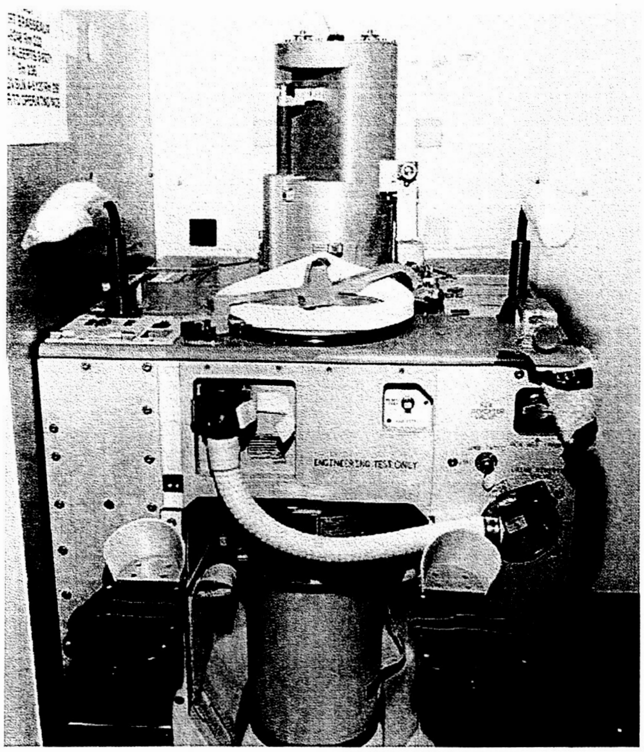

Figure 3. Existing waste collection hardware, the United States International Space Station Waste Collection System (US ISS WCS). Fiberglass canister beneath seat collects waste in bags.

The US ISS Waste Collection System, shown in Figure 5, has been flight tested on Shuttle and is slated for use on the International Space Station. It collects feces and toilet paper in porous PTFE/polyester bags; these bags are compressed into a removable, fiberglass canister. (See Figure 5; each canister holds approximately 25 bags.) By coincidence, Teflon membranes are the basis for a product used in the pharmaceutical industry for sterile lyophilization. A container incorporating an expanded ePTFE (ePTFE) membrane permits passage of water vapor, but not liquid water or bacteria. Thus a liquid sample can be frozen, dried, and subsequently stored while remaining sealed within a sterile container.

Hydrophobic membranes such as the WCS bags and ePTFE containers used in lyophilization present a barrier to liquid water because of water's surface tension. These fluorinated polymers have lower surface energy than water, so they resist wetting. The water surface at the entrance to a pore behaves like the skin of a balloon, stretching as the pressure driving water into the pore increases. Above a certain pressure, known as the breakthrough pressure, this surface tension force is overcome by the pressure force, and water leaks through the membrane. Breakthrough pressure is inversely proportional to pore diameter. For a 0.2 μm ePTFE membrane, it is approximately 10 psi. The current WCS bags have larger pores, and liquid weeps through the bags after they are compressed into the canister.

During drying, the porous membrane presents an added resistance to vapor flow, reducing energy efficiency. Smaller pores are more costly in this respect. When choosing a membrane material, this energy cost must be weighed against the safety benefits of higher breakthrough pressure and more reliable microbial containment.

SYSTEM DESIGN

OVERVIEW – The current prototype, shown in Figure 4, implements the TEC configuration described in Figures 1 and 2 in a package tailored to this application. The cylindrical chamber is approximately the same size as the current WCS collection canister it is intended to replace. Rather than being compacted into the canister, a bag of waste would be directed into one of the two holding cups of the lyophilizer. This allows immediate freezing of waste, minimizing biological activity and evolution of unwanted volatiles.

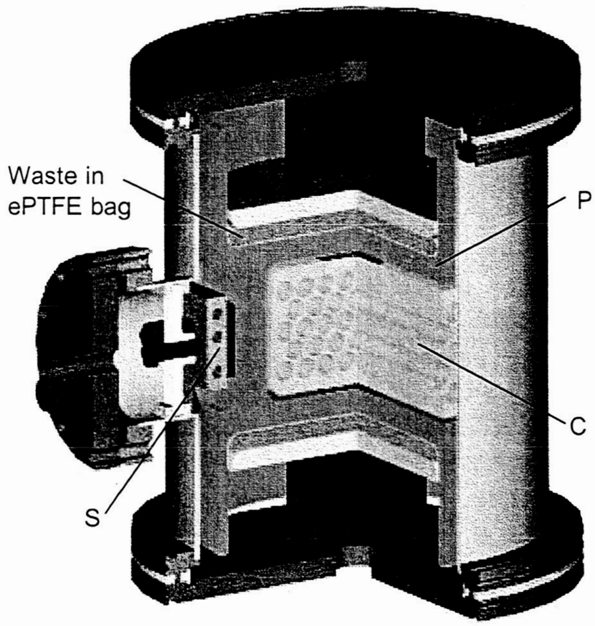

Figure 4. Pilot-Scale Thermoelectric Lyophilizer, 10" nominal diameter. Heat sink S, heating plate P, and condenser C correspond to components in Figures 1 and 2.

The heating plate P is continuously maintained at a low temperature. The condenser C is kept below 273K, and ice collected from the previous batch of waste remains on the condenser until both dried bags of waste have been replaced with new bags. As the new bags freeze, heat is pumped from P to C by running the TECs connecting them in reverse. This thaws the ice as the new waste freezes, saving time and energy.

HEAT LEAKS – Because the unit is intended to remain at low temperature between uses, minimizing heat flow from the vacuum chamber to the interior components is a high priority. Maintaining system vacuum addresses convective heat transfer. Reducing conductive heat transfer requires suspending the heating plate within the chamber using parts with small cross sectional area, made of material with low thermal conductivity. Radiative heat transfer varies with the fourth power of temperature, and is usually negligible in low-temperature processes. But if conduction and convection are reduced sufficiently, it becomes significant.[6]

Initial tests of the unit shown in Figure 4 led to design modifications aimed at reducing conductive and radiative heat leaks. Figure 5 shows the unit with lids removed. An aluminized Mylar liner has been added to the chamber walls to reflect radiated heat.

Figure 5. Unit with lids removed. Vacuum chamber lined with Mylar.

Figure 6 shows the interior components removed from the vacuum chamber. The nylon set screws protruding from the rims of the cups suspend the interior components within the chamber. The only other points of contact with ambient temperature surfaces are power and instrumentation wires, and fluid lines for coolant and water recovered from the condenser.

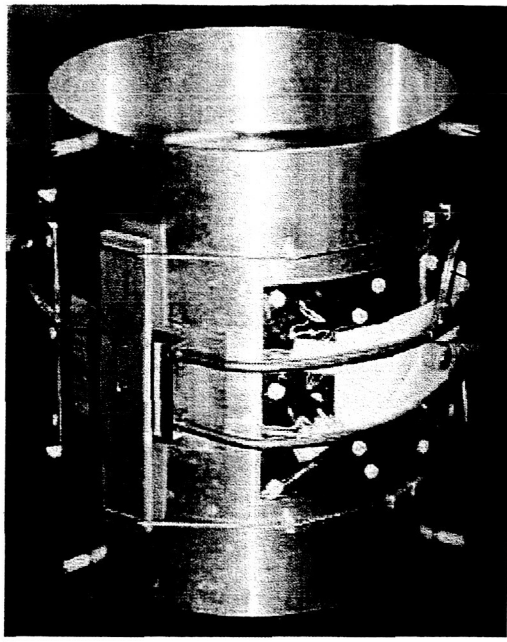

Figure 6. Interior components removed from vacuum chamber.

An additional modification is seen in Figure 6. The gold-plated heat sinks and associated are compressed against the sides of the heating plate by flexible aluminum bars and stainless steel cables. Thermal expansion causes significant changes in component dimensions during operation, yet TEC mounting pressure must remain within specifications to maintain good thermal contact while avoiding damage to TECs. To meet both requirements, mounting hardware is designed maintain the proper force by deforming elastically – in this case, the aluminum bars bend. The cables are used rather than screws to lengthen and constrict the heat transfer pathway between the heating plate and the higher temperature heat sink.

The previous laboratory-scale lyophilizer used stainless steel screws and insulating washers to compress TECs between system components. Experiments revealed significant heat leakage through the screws. When designing this unit, emphasis was placed on devising means to compress TECs within their specified pressure range without screws or other hardware. Ideally, the only points of contact between parts at different temperatures should be the TECs themselves.

Figure 7 shows the heating plate, condenser, and heat sinks, connected by TECs. A condenser that deforms elastically can be mounted within the heating plate and allowed to expand, holding itself in place and supplying the proper force to the TECs. Also, the condenser is the coldest part in the system; sheltering it within the slightly warmer heating plate reduces radiative heat transfer to the condenser. This is particularly important because the ice that forms on condenser surfaces is effectively a black body – it absorbs much more infrared radiation than low emissivity surfaces such as polished metals.

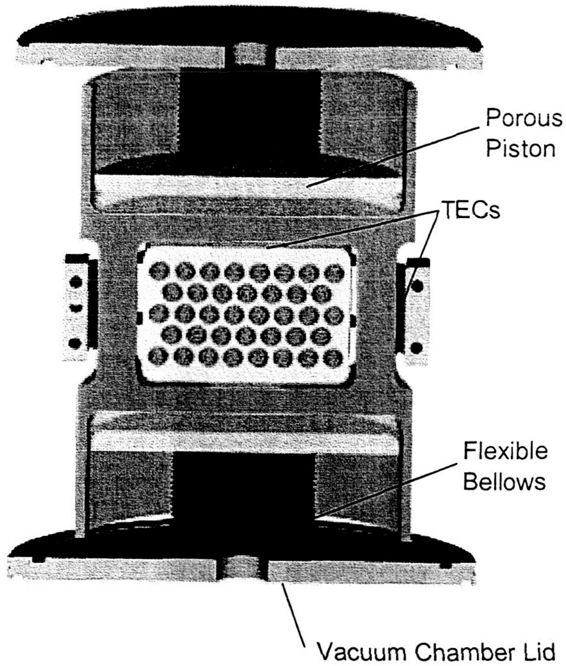

Figure 7. Areas of contact between system components.

The porous pistons compress the bags of waste against the heating plate. With the interior of the bellows exposed to ambient pressure, evacuating the chamber extends the bellows and imparts a force to the pistons. Refer to the Compression section below for a more detailed discussion.

FACILITATING HEAT TRANSFER – The TECs connecting the heat sink to the heating plate remove heat from the plate to maintain its temperature. Most of this heat is produced by the other set of TECs connecting the heating plate to the condenser. The rate at which heat is removed from the condenser equals the rate at which it flows to the waste. But resistive heating of the TECs releases additional heat at a rate equal to the electrical power delivered to the TECs. This heat flows through the heating plate and is pumped out to the heat sinks. Resistance to heat flow within the heating plate will reduce energy efficiency. The shape and material of the heating plate and configuration of the TECs should minimize this heat transfer resistance while fulfilling practical system requirements.

Figure 8 shows one eighth of the heating plate. (The plate has three axes of symmetry, so only one eighth is required for analysis.) During drying, heat flows in from condenser TECs, out to waste, and out to heat sink TECs. If condenser TECs are positioned as shown in Figure 7, a temperature difference of 2-3 K develops within the heating plate under typical drying conditions, using four smaller condenser TECs, one on each side of the condenser, would allow waste heat from the condenser TECs to travel a more direct route to the heat sink, reducing this source of inefficiency. See the Condenser section below for details.

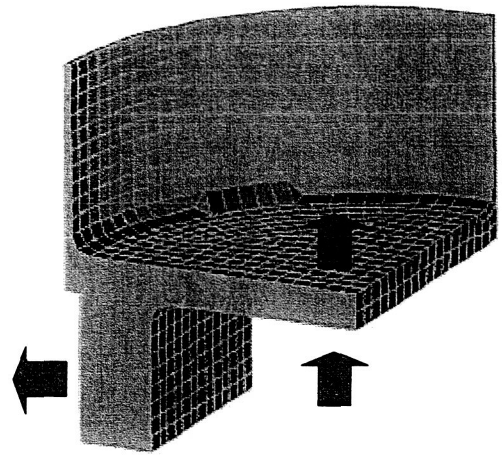

Figure 8. Finite element mesh of heating block. Arrows indicate heat sources and sinks.

SEMIPERMEABLE BAGS – Figure 9 shows one of the porous bags use on the WCS. The ring at the opening peels off, exposing adhesive used to seal the bag. A porous bag is used so that air can be drawn through it during use, directing waste into the bag. Holes at the corners hold the bag in place under the seat.

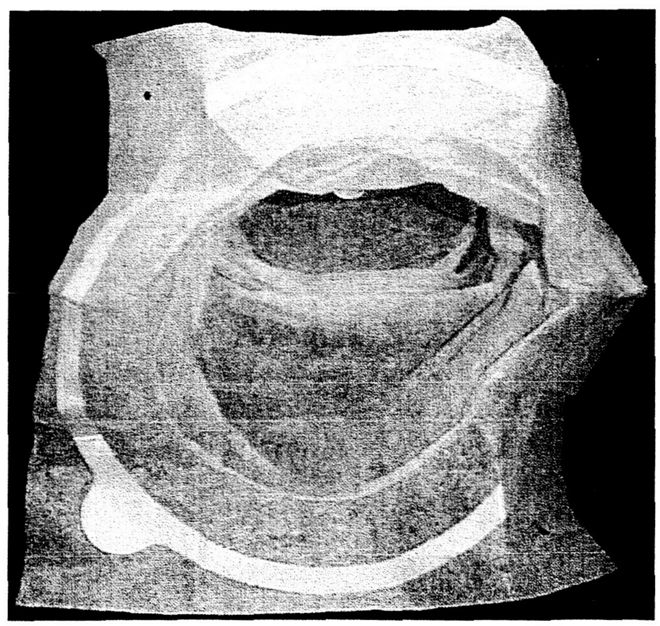

Figure 9. Waste collection bag for use with US ISS WCS. Non-woven polyester with PTFE coating.

Figure 10 is an electron micrograph of ePTFE. The material is manufactured by repeatedly stretching sheets of PTFE to create small tears. Different stretching techniques produce different pore sizes and characteristics. Thin ePTFE membranes have low mechanical strength, and are generally mounted to a supporting fabric such as non-woven poypropylene.

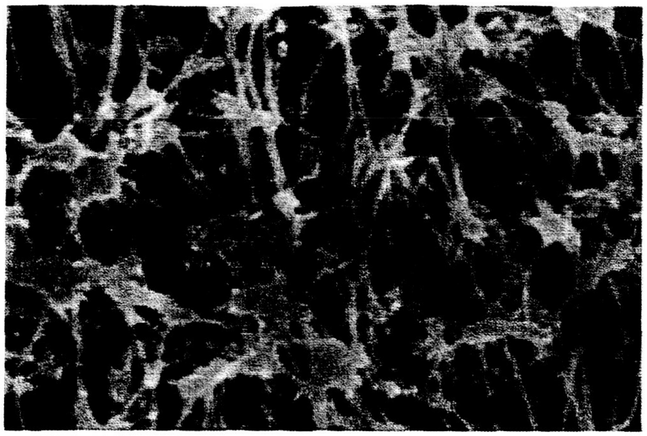

Figure 10. Expanded polytetrafluoroethylene (ePTFE) membrane, 0.2 μm nominal pore size.

Lyophilizing waste within bags adds two additional resistors to the circuit in Figure 2: heat must flow in through the bag from heating plate to waste, and water vapor must flow out through the bag and into the vacuum chamber. But containing the waste in bags throughout the process offers pronounced practical advantages: simplified material handling, compatibility with existing hardware, biological safety, and reliability. Even in a case of lyophilizer failure, bagged waste can be removed and stored with minimal risk of contamination. If a bag breaks, waste can be wiped up, and the broken bag and wipes placed in another bag to be lyophilized.

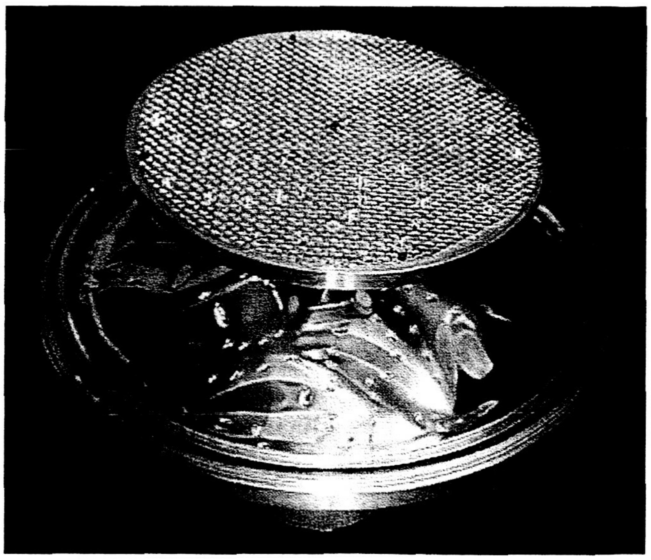

Figure 11. Porous piston, current implementation. Stainless steel screen compresses bag of waste.

COMPRESSION – Figure 11 shows a vacuum chamber lid and porous piston. The piston is a stainless plate with holes drilled through it and a coarse mesh stainless steel screen attached to the surface that compresses the bag of waste.

Figure 12 shows a side view. During drying, the lid is at ambient temperature and the piston contacts the frozen waste. Heat flow from lid to piston would increase drying rate, but this heat would increase the load on the heat sink TECs. To maximize energy efficiency, heat flow from lid to piston should be minimized.

In the current implementation, this is accomplished by isolating the piston from the bellows assembly. A stainless steel acorn nut in the center of the piston rests against the blank flange at the end of the bellows. Both of these parts are stainless steel, which has a relatively high ratio of compressive strength to thermal conductivity, so a small contact area transmits large forces but small amounts of heat. The nylon screws at the perimeter of the flange fit loosely in holes in the flange, centering the piston on the flange. This arrangement allows the piston to pivot, preventing the piston from imparting torque to the bellows if the if the bag of waste does not compress evenly.

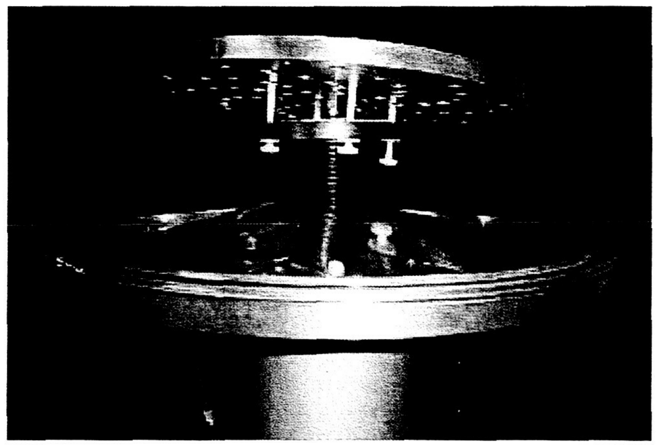

Figure 12. Porous piston, side view. Nylon screws center piston on bellows assembly. Stainless steel acorn nut is only point of contact between piston and bellows, transmitting required force while minimizing heat transfer.

The force needed to compress the bags can be developed by evacuating the chamber while exposing the interior of the bellows to atmospheric pressure, resulting in a pressure on the bag of waste equal to 1 atm times the ratio of bellows area to piston area.

Previous compression experiments demonstrated improved drying rate and energy efficiency when skim milk was compressed at approximately 5 psi. When the porous layers of dried material are compressed, resistance to heat transfer decreases and resistance to water vapor transfer increases.[7] Presumably there exists an optimal pressure for each material that balances these effects.

To estimate the optimum compression force, and thus the optimum bellows size, experiments with appropriate wastes at varying pressures are required. As seen in Figure 12, pneumatic cylinders have been connected to the lids to conduct these experiments. The cylinder rods connect to the flanges at the ends of the bellows. A regulator provides air at known pressure to the cylinders, and resistive position sensors in the cylinders monitor the position of the rods.

The unit has been tested at pressures of up to 14 psi, meaning 14 psi is the pressure on the bag of waste. This translates to 700 lbf total on each 8" diameter piston. Observed plastic deformation of the interface between acorn nut and bellows flange was minimal. Note that a design with two pistons per chamber results in piston forces that offset. This enables the heating plate to be mounted in the vacuum chamber using a minimum of hardware, reducing heat leaks.

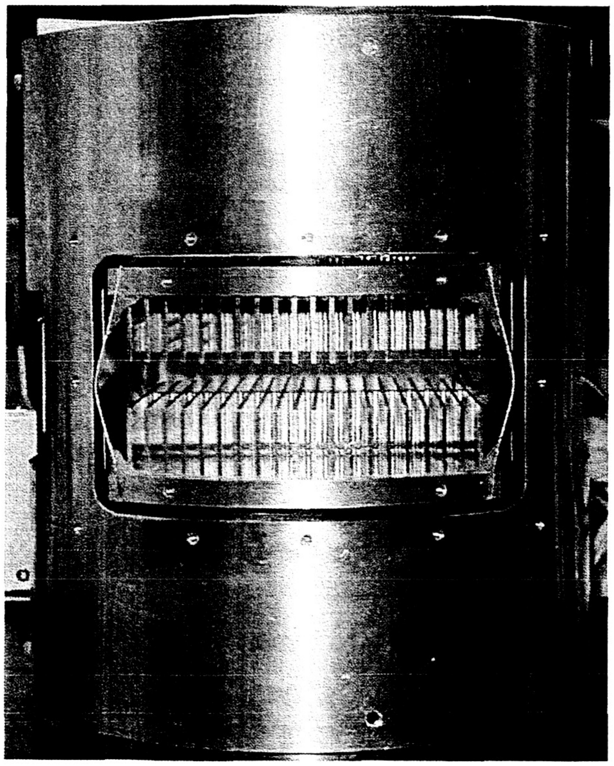

Figure 13. Photo of temporary condenser mounted in heating plate.

CONDENSER – Condenser design has not been finalized. Figures 4 and 7 show a design intended for use in microgravity. Figure 13 shows a simpler, temporary condenser that requires gravity for liquid water recovery. The condenser consists of identical finned plates connected by aluminum springs. Figure 6 shows the condenser after assembly. Mylar covers the gap between condenser and heating plate to prevent ice formation on external condenser surfaces. One end of the condenser is sealed with ePTFE. When ice is melted for recovery, this end is positioned at the bottom. Water pools at the closed end and is extracted through the tube visible in Figure 6.

The thermal conductivity of ice, while approximately an order of magnitude higher than that of typical lyophilized solids, is low enough that a thick layer of ice at the condenser presents a significant heat transfer resistance. Ice layer thickness can be minimized by maximizing condenser surface area. But long, narrow passageways restrict vapor flow and cause uneven ice deposition. The condenser shown in Figure 13 exhibits this problem, and the resulting heat transfer resistance reduces energy efficiency and increases drying time. Replacing this component promises to increase system performance dramatically. The ideal condenser should 1) transfer heat to the heating plate efficiently (e.g., with TECs on four sides rather than two, 2) maintain mounting pressure on TECs within specifications, 3) minimize ice layer thickness while allowing ice to form evenly, and 4) contain liquid water for collection with or without gravity.

SUMMARY – Figure 14 shows the assembled prototype and ancillary hardware. The current vacuum pump at right, and chiller and power supplies at left, are laboratory-style equipment chosen for convenience rather than minimum equivalent system mass.

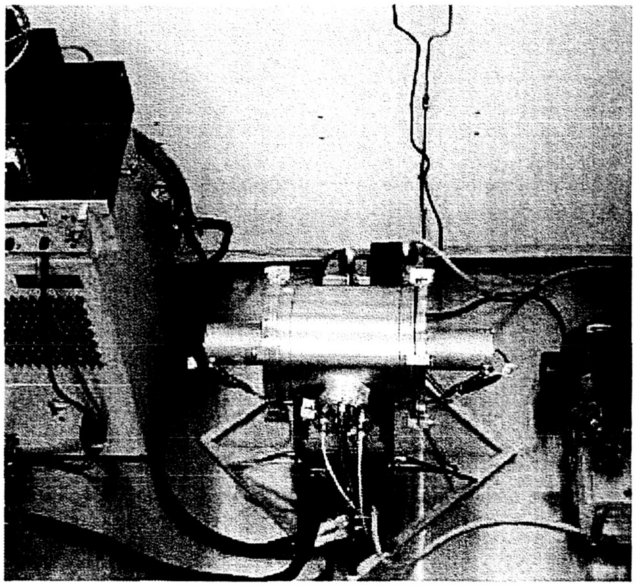

Figure 14. Assembled prototype including vacuum pump (right), chiller and power supplies (left). Chamber rotates in stand so that either end may face up.

The chiller supplies cooling water to the heat sinks at 40 Deg. F (8 Deg. C), under the assumption that water at this temperature is available as a utility on the ISS or future spacecraft. In contrast to typical lyophilizers, the vacuum pump does not run continuously. It evacuates the chamber initially, then remains off during drying unless abnormally high pressure is detected in the chamber.

PERFORMANCE

PRODUCT QUALITY – The lyophilizer has been tested with bags of water, skim milk, and monkey feces. Average water recovery from three runs with monkey feces was 98%. Lyophilized samples were dried in a vacuum oven to determine residual water content. Dried product contained an average of 5% water and other volatiles by weight.

ENERGY EFFICIENCY – Drying rate and power consumption data indicate that resistance to heat and mass flow from heating plate to condenser (resistors 1-4 in Figure 2) is large and increases with time. The increasing resistance is consistent with observed ice formation on the condenser. Ice forms at the condenser opening, preventing vapor flow into the interior. A different condenser design is required.

High- or low-temperature reactors require good insulation to operate efficiently. This is particularly true in the case of small reactors, with high surface area to volume ratios. As discussed above, the prototype was designed to minimize heat transfer between low-temperature interior components (heating plate and condenser) and ambient-temperature components (vacuum chamber and pistons/bellows). In this respect the prototype performs better than anticipated. When drying is complete, ice remains on the condenser and dried bags remain compressed by the pistons. The unit idles in this state until new waste arrives. In this condition, total electrical power required by the TECs to keep the heating block and condenser below 273K is 4W.

CONCLUSIONS

Previous research has demonstrated that lypohilization can be adapted to recover water from mixed liquid/solid wastes, and that a lyophilizer can be built using TECs instead of traditional heat pumps. Building on this research, a practical prototype has been developed to process these wastes in the context of long-term human space missions. The prototype uses porous bags to contain waste throughout the process, simplifying material handling in microgravity. It compresses these bags during drying to improve drying rate and energy efficiency. Tests showed that low-temperature Internal components were well insulated from the ambient-temperature vacuum chamber. Bags of feces were successfully dried to 5% water content, with 98% of available water recovered.

ACKNOWLEDGMENTS

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DEFINITIONS, ACRONYMS, ABBREVIATIONS

VARIABLES

$\mathbf{Q}_c$: Heat Removed from Cold Reservoir [W]

$\mathbf{T}_c$: Temperature of Cold Reservoir [K]

$\mathbf{T}_h$: Temperature of Hot Reservoir [K]

W: Power Consumed by Heat Pump [W]

LOCATION SUBSCRIPTS

C: Condenser

P: Heating plate

R: Room (ambient air)

S: Heat sink

ACRONYMS

COP: Coefficient of Performance

ePTFE: Expanded Polytetrafluoroethylene

ESM: Equivalent System Mass

EVA: Extravehicular Activity

ISS: International Space Station

TEC: Thermoelectric Cooler

WCS: Waste Collection System